

## PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): Venkat Selvamanickam, et al.

Title: METALORGANIC CHEMICAL VAPOR DEPOSITION (MOCVD)  
PROCESS AND APPARATUS TO PRODUCE MULTILAYER HIGH-  
TEMPERATURE SUPERCONDUCTING (HTS) COATED TAPE

App. No.: 10/602,468

Filed: June 23, 2003

Examiner: Jennifer C. McNeil

Group Art Unit: 1775

Customer No.: 34456

Confirmation No.: 2661

Atty. Dkt. No.: 1014-SP156-US

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MS AF

Commissioner for Patents

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## DECLARATION UNDER 37 C.F.R. §1.132

Sir, I hereby declare and state:

1. I am a joint inventor of the subject matter presently claimed in the above-identified patent application.

2. I received my doctorate degree in Materials Engineering from the University of Houston in Houston, TX.

3. I have been employed by IGC/SuperPower, Inc. since 1994, wherein I have been mainly engaged in research and development of superconducting materials, superconducting conductors, and processes for forming same.

4. I have reviewed the Office Action dated October 18, 2005, including the positions taken by the PTO with respect to several prior art references. I have also particularly reviewed the subject matter of US 5,106,828, Bhargava et al. (Bhargava). For the reasons discussed below, Bhargava fails to disclose (or suggest) all features of the claimed invention.

5. The claimed invention is drawn to a superconductive article comprising a substrate tape and a superconductive layer. The superconductive layer notably includes a plurality of superconductive films of the same material, the films being in direct contact with each other. As described in the present specification, the films are formed by a metalorganic chemical vapor deposition (MOCVD) process, in which metalorganic precursors are reacted with each other in a deposition chamber, the reaction product forming a superconductive material that deposits on the substrate tape. As described in the present specification, pages 23+ in connection with FIGs. 1-4b, the substrate tape is translated through an MOCVD system containing multiple compartments arranged in series, each defining a deposition zone (see Zones A-E). Each zone has associated unique control parameters as described in Tables 1-5. As the substrate translates through the MOCVD system, the substrate tape experiences multiple deposition events, each deposition event corresponding to each zone, thereby forming an identifiable, discrete superconducting film. That is, by passage of the tape through a zone, the zone forms as-deposited superconductive material in the form of a film.

Attached hereto is an SEM micrograph showing a superconductive layer produced by coating of 3 three superconductive films corresponding to YBCO1, YBCO2, and YBCO3. Analysis shows that the individual films are identifiable and are separated by each other by boundary regions corresponding to the arrows shown on the attachment.

6. In contrast, Bhargava is drawn to a solution/sol-based process flow in which the material deposited on the substrate is a sol, corresponding to an organic *precursor* of the superconductive material. In sol-based processing, given the rheology of the sol, it is necessary to form a first precursor layer, followed by drying and subsequent deposition of a second precursor layer. Sol deposition followed by drying is repeated until the desired thickness of a precursor of a superconductive layer is deposited. Upon achievement of the desired thickness, the entire layer is heated, generally in an oxygen-containing environment to convert the precursor material into a superconductor oxide material.

At no time during the process flow of Bhargava are multiple superconductive films formed on top of each other; rather, multiple *precursor* films formed. Upon subsequent heat treatment and conversion of the precursor films, the films form a single, unitary layer having no

identifiable film boundaries. That is, due to the process flow associated with sol-based processing, identifiable superconductive films are not formed. This is due to the fact that conversion of the deposited precursor layers takes place in a single heat treatment step, while the MOCVD process according to embodiments of the present invention result in as-deposited superconductive films formed via multiple, discrete depositions steps corresponding to respective zones.

Attached is an SEM cross-section of a sol-based film formed by metalorganic deposition (MOD) that was formed by multiple deposition and drying process steps to build-up the layer thickness shown in the micrograph, then converted by heat treatment. The SEM cross-section is representative of the teachings of Bhargava. As shown, the superconductive layer is a unitary structure composed of a single, monolithic mass of material entirely devoid of individual films.

7. In summary, it is quite clear that sol-based processing having intermediate drying steps cannot result in an superconductive layer having multiple, identifiable superconductive films.

8. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

February 21, 2006

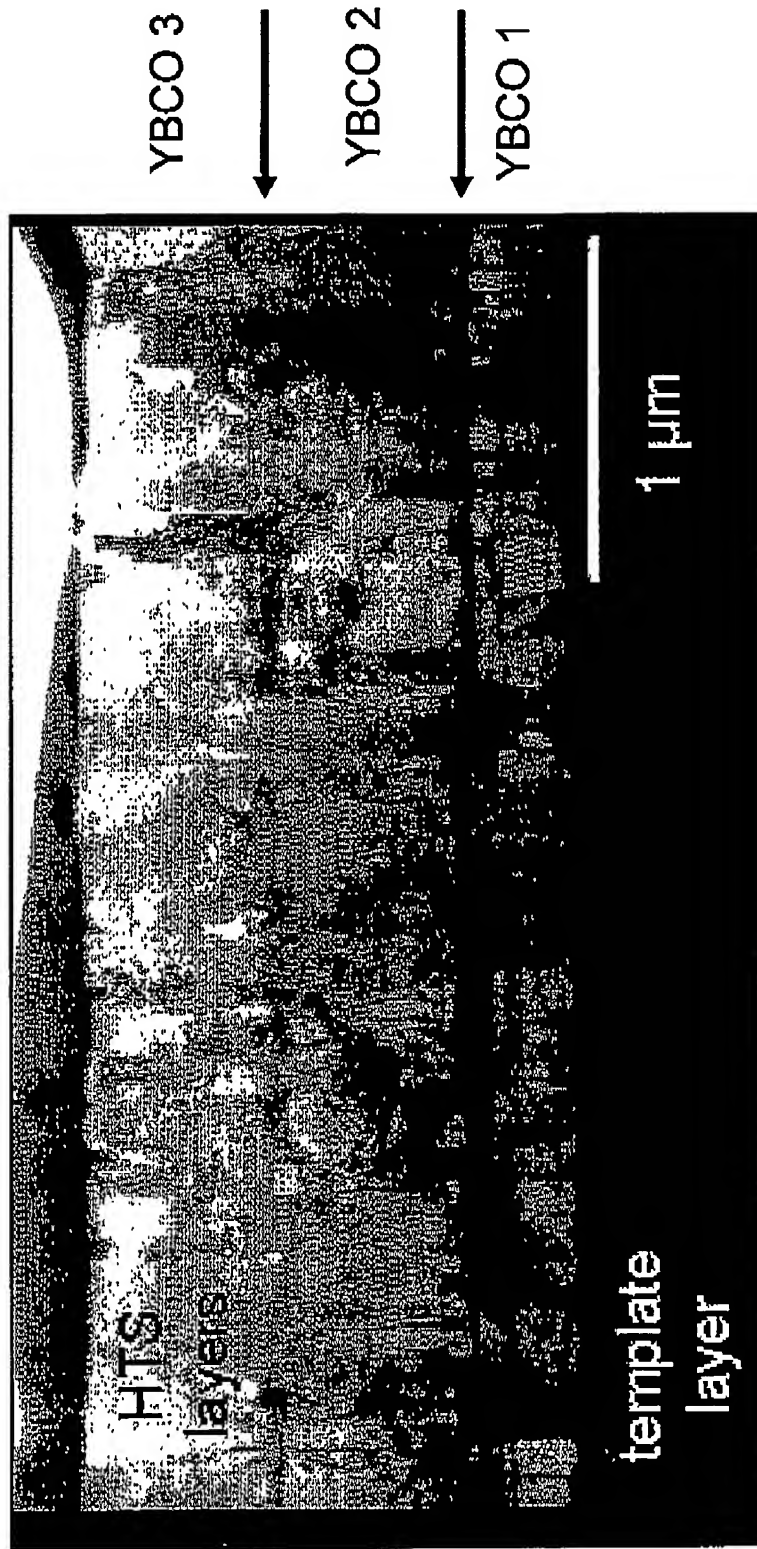
Date

Respectfully submitted,



Venkat Selvamanickam

# YBCO produced by MOCVD in 3 passes



Arrows show the boundaries between the 3 layers.

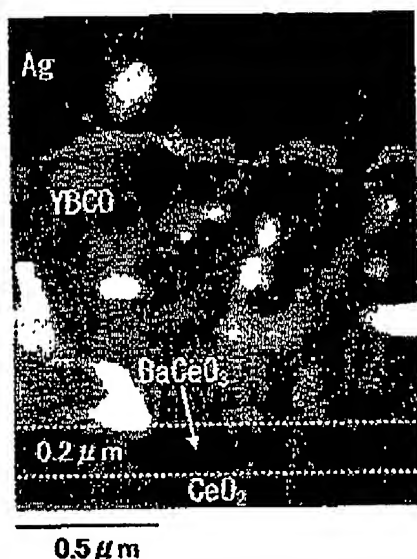


Fig. 2. TEM photograph of the cross section in the four times coated film with 1.1  $\mu\text{m}$  thick.

thickness exists at the interface between the YBCO and  $\text{CeO}_2$ . The reason for the degradation of  $J_c$  in the thin film could be explained by the existence of the  $\text{BaCeO}_3$  layer.

#### 4. Long tape processing

When we applied the TFA process to the long tape production, the design of the gas flow system becomes a key technology. Fig. 3 shows the position dependence of the  $J_c$  in the transverse and parallel gas flow to the long direction of the tape. According to this result, a homogeneous tape can be fabricated by the transverse gas flow system. On the other hand, the  $J_c$  values in the parallel gas flow system drastically decrease from the windward to the leeward. In order to understand this tendency, we investigated the growth rate of YBCO phase. Fig. 4 shows the position dependence of the growth rate in the parallel gas flow system. From this result, the growth rate of YBCO decreased with increasing the tape length. Therefore, the YBCO reaction in the parallel gas flow

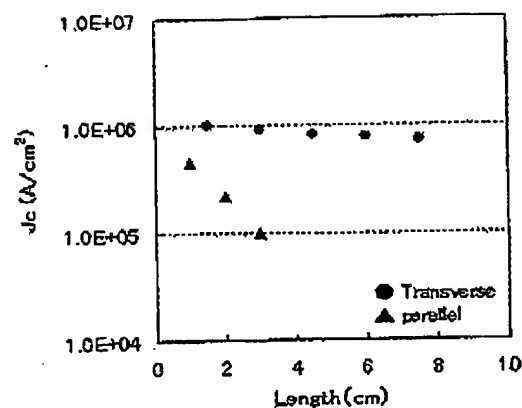


Fig. 3. Position dependence of the  $J_c$  in the transverse and parallel gas flow to the long direction of the tape.

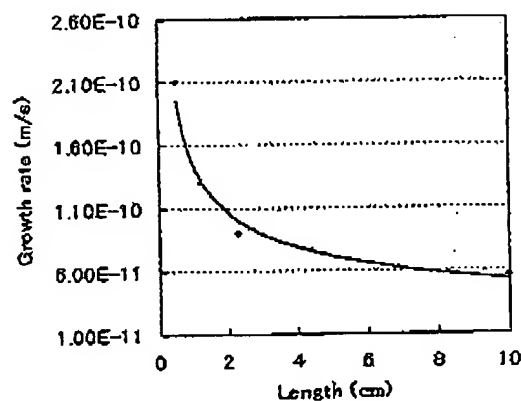


Fig. 4. Position dependence of the growth rate in the parallel gas flow system.

system may not be completed within the same annealing time. The reason for this phenomenon could be explained by the calculation of two-dimensional analysis for the mass transfer of  $\text{H}_2\text{O}$  and HF gases in the gas boundary layer ahead of the surface of the precursor. The details will be published in another report [18]. Concerning the long tape processing, the above tendency should be an important problem to be solved a reasonable production rate in the continuous system. Therefore, in order to obtain a high production rate, the